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Indian Standard

CHEMICAL ANALYSIS OF FERROCHROMIUM

PART 2 DETERMINATION OF SILICON IN LOW CARBON FERROCHROMIUM BY GRAVIMETRIC METHOD

UDC 669·15·26-198 : 543·21 [546·28]

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BUREAU OF INDIAN STANDARDS
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NEW DELHI 110002

July 1992 **Price Group 1**

Methods of Chemical Analysis of Ferrous Metals Sectional Committee, MTD 2

FOREWORD

This Indian Standard (Part 2) was adopted by the Bureau of Indian Standards, after the draft finalized by the Methods of Chemical Analysis of Ferrous Metals Sectional Committee had been approved by the Metallurgical Engineering Division Council.

IS 1559 : 1961 'Methods of chemical analysis of ferroalloys' covers the chemical analysis of different ferroalloys, namely, ferrosilicon, ferrochromium, ferromanganese and speigeleisen, silicomanganese, ferromolybdenum, ferrophosphorus, ferrotitanium, ferrovanadium and ferrotungsten. While reviewing this standard, the Sectional Committee had decided that a separate series for each ferroalloy be prepared. Accordingly, IS 1559 in its various parts was published for the chemical analysis of ferrosilicon.

The chemical analysis of ferromolybdenum is covered in a series of parts of IS 12614. The chemical analysis of ferrochromium is covered in a series of parts of this standard. With the publication of these parts the analysis of relevant constituent prescribed for ferrochromium in IS 1559 : 1961 will be superseded. This part covers the method for determination of silicon in low carbon ferrochromium. The other parts are:

Part 1 Determination of silicon in high carbon ferrochromium
Part 3 Determination of phosphorus in low carbon ferrochromium
Part 4 Determination of total sulphur in low and high carbon ferrochromium
Part 5 Determination of chromium in high carbon ferrochromium
Part 6 Determination of chromium in low carbon ferrochromium
Part 7 Determination of nitrogen in high and low carbon ferrochromium
Part 8 Determination of carbon (total) in low and high carbon ferrochromium

In this revisoin the determination of silicon in low carbon ferrochromium has been updated.

In reporting the result of a test or analysis made in accordance with this standard, if the final value observed or calculated is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

Indian Standard

CHEMICAL ANALYSIS OF FERROCHROMIUM

PART 2 DETERMINATION OF SILICON IN LOW CARBON FERROCHROMIUM BY GRAVIMETRIC METHOD

1 SCOPE

This standard (Part 2) describes the method for determination of silicon in low carbon ferrochromium (containing carbon up to one percent).

2 REFERENCES

The following Indian Standards are necessary adjuncts to this standard:

IS No.	*Title*
265 : 1987	Hydrochloric acid (*third revision*)
266 : 1977	Sulphuric acid (*second revison*)
1070 : 1992	Reagent grade water — Specification (*third revision*)
1472 : 1977	Methods of sampling of ferro-alloys for determination of chemical composition (*first revision*)

3 SAMPLING

Samples shall be drawn and prepared in accordance with IS 1472 : 1977.

4 QUALITY OF REAGENTS

Unless specified otherwise, analytical grade reagents and distilled water (*see* IS 1070 : 1992) shall be empolyed for the test.

5 DETERMINATION OF SILICON

5.1 Outline of the Method

After dissolution of the sample, silicic acid is dehydrated by fuming with perchloric acid. The solution is filtered and the silica is ignited, weighed and then volatilized with hydrofluoric acid. The residue is ignited and weighed, the loss in weight represents silica.

5.2 Reagents

5.2.1 *Hydrochloric Acid*, rd = 1·16 (conforming to IS 265 : 1987)

5.2.2 *Perchloric Acid*, 60 percent

5.2.3 *Hydrofluoric Acid*, 40 percent

5.2.4 *Dilute Sulphuric Acid*, 50 percent

5.2.5 *Silver Nitrate Solution*, 2 percent (*m/v*)

5.3 Procedure

5.3.1 Weigh accurately 1·00 g of sample into a 400 ml glass beaker, add 20 ml of hydrochloric acid and heat until effervescence ceases and sample dissolves completely.

5.3.2 When the beaker has cooled down add 20 to 25 ml of perchloric acid (60 percent) and heat to fuming and reflux for 15 minutes and cool. Add 2 to 3 ml of hydrochloric acid and dilute with distilled water. Boil and filter the silica through ashless, medium textured filter paper. Rinse the beaker with warm water and transfer the contents to the filter.

5.3.3 Wash the residue with warm hydrochloric acid and then finally wash several times with hot water, till it is free from chloride.

NOTE — Check the filtrate for presence of chloride using silver nitrate solution (2 percent).

5.3.4 Transfer the residue (**5.3.3**) alongwith the filter to a platinum crucible. Evaporate to dryness and ignite at 1 100°C in a muffle furnace to constant mass. Cool in a desiccator and weigh the crucible (A).

5.3.5 Moisten the impure silica (**5.3.4**) with a few drops of water. Add about 10 ml of hydrofluoric acid and 2 or 3 drops of dilute sulphuric acid (50 percent) and cautiously evaporate to dryness. Ignite at 1 100°C to a constant mass. Allow to cool in a desiccator and weigh the crucible (B).

5.3.6 *Blank*

Carry out a blank determination following the procedure specified in **5.3.2** to **5.3.5** and using same amount of all reagents. Correct the final results for the blank value, if any (C).

5.4 Calculation

$$\text{Silicon, percent by mass} = \frac{[(A - B) - C] \times 46{\cdot}74}{D}$$

where

A = mass in g, of crucible before hydrofluorization,

B = mass in g, of crucible after hydrofluorization,

C = mass in g, of silica from the blank, and

D = mass in g, of sample taken.

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